INCH-POUND

MIL-P-14067B (AR) AMENDMENT 4 8 February 1999 SUPERSEDING AMENDMENT 3 15 November 1971

# **MILITARY SPECIFICATION**

# POWDERS, METAL, ATOMIZED (FOR USE IN AMMUNITION)

MIL-P-14067B was inactivated after 20 February 1998 for new design.

This Amendment forms a part of Military Specification MIL-P-14067B (MU), dated 10 March 1967, and is approved for use by the US Army Research, Development and Engineering Center, and is available for use by all Departments and Agencies of the Department of Defense.

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- \* 1.2 <u>Classification</u>. Add the following type:
  - "Type V Aluminum powder, Atomized for use in the rocket motor for the M913."
- \* 2. <u>Applicable Documents</u>. Delete in its entirety and substitute the following:
  - "2. Applicable documents.
  - 2.1 General. The documents listed in this section are needed to meet the requirements specified in sections 3 and 4 of this specification. This section does not include documents in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all requirements documents cited in sections 3 and 4 of this specification, whether or not they are listed.

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## 2.2 Government documents.

2.2.1 <u>Specifications, standards, and handbooks</u>. The following specifications, standards, and handbooks of the exact revision below form a part of this document to the extent specified herein.

# **STANDARDS**

#### DEPARTMENT OF DEFENSE

MIL-STD-129 - Marking for Shipment and Storage
 MIL-STD-1233 - Procedures for Determining Particle Size, Particle Size Distribution and Packing Density of Powdered Materials
 MIL-STD-1234 - Pyrotechnics, Sampling, Inspection and Testing

MIL-STD-1916 - DOD Preferred Methods for Acceptance of Product

2.2.2 Other Government documents, drawings and publications. The following other Government documents, drawings and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the solicitation.

# Code of Federal Regulations

49 CFR 71-90 - Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles.

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Officer, Washington, DC. 20402. Orders for the above Publications should cite: 49 CFR 71-90 (latest revision).)

2.3 <u>Non-Government publications</u>. The following document(s) form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation (see 6.2).

# **PUBLICATIOINS**

American Society for Testing and Materials

ASTM E 34-58 - Chemical Analysis of Aluminum and Aluminum Base Alloys

(Application for copies should be addressed to the American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19428.)

- 2.4 <u>Order of precedence</u>. In the event of a conflict between the text of this document and the references cited herein, except references to higher level program unique specifications for this program, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained. (See contract provisions for additional precedence criteria)."
- \* 3.1.1 Add type V to the title and the text.

# PAGE 4 \* 3.2 Delete Table I in its entirety and substitute New Table I as follows:.

"Table I. Chemical and Physical Properties

	Ţ	II	III	IV	V	
	Magnesium	Aluminum	65/35	Ellipsoidal	Aluminum	Test
	Powder	Powder	Mg/Al	Powder	Powder	Para.
			Alloy			
REQUIREMENT			Powder			
Magnesium, % min	98.0		65 <u>+</u> 5	98		4.5.5
Aluminum, % min		98.75	$35 \pm 5$		99.00	4.5.10
Volatile material at	0.1	0.1	0.1	0.1	0.1	4.5.2
105 deg C, % max						
Oil and grease,		0.2	0.2		0.2	4.5.8
% max						
Zinc, % max	1.5	0.3		0.5		4.5.6
Silicon, % max		0.25	0.2			4.5.9
Alloy as Iron,	0.05	0.55	0.1	0.05	0.20	4.5.4
% max						
Carbides, % max	0.004			0.004		4.5.3
Other impurities,	0.3			1.0		4.5.8
% max						
Total Al and Mg,			98.0			4.5.12
% max						
Apparent Density.	0.95	1.0	1.0	0.95		4.5.14"
G/ml (min)						

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# 3.3. Granulation, Table II, Type IV, Ellipsoidal Magnesium:

Add the following granulations:

44		Percent Retained on	
		Type IV 30/50	Type IV 20/50
	Type I 30/40	Ellipsoidal	Ellipsoidal
U.S.S Sieve No.	Atomized	Magnesium	Magnesium
20	0.0 - 0.1  max	0.0 - 0.1  max	0.0 - 0.1  max
30	0.0 - 7.0  max	0.0 - 7.0  max	0.0 - 16.0  max
40	43 - 53	43 - 53	45 - 65
50	40 - 56	40 - 56	20 - 35
60	0 - 3.0	0 - 3.0	0 - 3.0
Thru 60	0 - 0.1	0 - 0.1	0 - 0.1"

PAGE 3

# Add Paragraph 3.4.1 as follows:

- \* "3.4.1 Particle diameter for type V. The average particle diameter for type V aluminum powder shall be  $25 \pm 5$  microns."
- \* 4.1. Delete in its entirety and substitute the following
  - "4.1 Classification of inspections.
    - a. Conformance inspection (see 4.2).

# Table IV. REQUIREMENTS/VERIFICATIOIN CROSS REFERENCE MATRIX

METHOD OF VERIFICATION	CLASSES OF VERIFICATION
N/A Not Applicable	A - Conformance inspection
1 – Analysis	
2 – Demonstration (end item test)	
3 – Examination	
4 – Test	

Section 3	Description	Verification			ion		Verification	Section 4
		Method			d		Class	
		N	1	2	3	4		
3.1	Shape				X		A	4.3.1
3.2	Chemical Properties					X	A	4.3
3.3	Granulation					Х	A	4.3.15
3.4	Average Particle Diam				X		A	4.3.13

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- \* 4.2 <u>Inspection provisions</u>. Delete in its entirety and substitute the following:
  - "4.2 Conformance inspection.
  - 4.2.1 <u>Inspection lot formation</u>. Lot formation shall be in accordance with MIL-STD-1916. In addition, the inspection lot shall consist of one lot of metal powder that has been produced by one manufacturer, in one unchanged process, using the same materials and methods, in accordance with the same specification revisions. All material submitted for inspection in accordance with this specification shall comply with the homogeneity criteria, regardless of the type of inspection procedure which is being applied to determine conformance with requirements.
  - 4.2.2 <u>Classification of characteristics</u>. There are three classes of characteristics covered in this document. These are critical characteristics, major characteristics and minor characteristics. (See MIL-STD-1916 for definitions of critical, major and minor classification of characteristics.). Unless otherwise specified herein or provided for in the contract, alternative conformance procedures, methods, or equipment, such as statistical process control, tool control, variables sampling or other types of sampling plans, may be used by the contractor when they provide, as a minimum, the level of quality assurance required by the provisions herein. Prior to applying such alternative procedures, methods, or equipment, the contractor shall describe them in a written proposal submitted to the Government for evaluation. When required, the contractor shall demonstrate that the effectiveness of each proposed alternative is equal to or better than the specified conformance provision(s) herein. In cases of dispute as to whether the contractor's proposed alternative(s) provides equivalent assurance, the provisions of this specification shall apply. All approved alternative provisions shall be specifically incorporated into the contractor's quality program or inspection system, as applicable.

# CONFORMANCE INSPECTION

# **CLASSIFICATION OF CHARACTERISTICS**

PARAGRAPH	TITLE			DRAWING NUMBER
		SHEET 1 OF	1	
4.2.2.1	Metal Powder			NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
Critical	None defined	CRITERIA	PARAGRAPH	REFERENCE
<u> </u>	none derried			
Major				
101	Shape	4.2.3.1	3.1	4.3.1
102	Volatile material	4.2.3.1	3.2	4.3.2
103	Carbides	4.2.3.1	3.2	4.3.3
104	Alloy iron as Fe	4.2.3.1	3.2	4.3.4
105	Free metallic magnesium	4.2.3.1	3.2	4.3.5
106	Zinc	4.2.3.1	3.2	4.3.6
107	Other impurities	4.2.3.1	3.2	4.3.7
108	Oil and grease	4.2.3.1	3.2	4.3.8
109	Silicon as Si	4.2.3.1	3.2	4.3.9
110	Free metallic aluminum	4.2.3.1	3.2	4.3.10
111	Percentage of aluminum	4.2.3.1	3.2	4.3.11
	and magnesium for			
	Type III powder			
112	Total aluminum and	4.2.3.1	3.2	4.3.12
	Magnesium for Type			
	III powder			
113	Average particle	4.2.3.1	3.4	4.3.13
	Diameter			
114	Apparent density	4.2.3.1	3.2	4.3.14
115	Granulation	4.2.3.1	3.3	4.3.15
116	Spectrochemical	4.2.3.1	3.2	4.3.16
	Analysis			
117	Shape of ellipsoidal	4.2.3.1	3.1	4.3.17
	Type IV			
Minor	None defined.			
111101				
NOTES:	<u> </u>	1	1	1

NOTES:

# CONFORMANCE INSPECTION CLASSIFICATION OF CHARACTERISTICS

PARAGRAPH	TITLE			DRAWING NUMBER
4.2.2.2	Drums, sealed	SHEET 1 OF 1		NEXT HIGHER ASSEMBLY
				ASSEMBLI
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
Critical	None defined			
Major				
101	Weight of Contents	Level II		Scale
102	Gasket missing or cover improperly closed	Level II	5.1	Visual
Minor				
201	Marking misleading or Unidentifiable	Level I	5.2	Visual
NOTES:				

# 4.2.3. Sampling for laboratory testing.

- 4.2.3.1 <u>Sampling of lot</u>. A portion of the stream of powder flowing into the drums shall be diverted into a gallon container. The portions shall be at such intervals so that a representative material from the bottom, middle and top portions of each drum is obtained. The sample in the gallon container shall be put through a 16 to 1 sample reducer and run through once to collect a five pound sample. The five pound sample shall then be run through a sample splitter to obtain two samples. If either of the samples fails to comply with any of the requirements specified, the lot shall be rejected.
- 4.2.3.1.1 <u>Sampling for granulation and particle size</u>. Each container shall be rolled and tumbled for ten minutes or until the metal powder is completely mixed in the container. Then a sample of approximately 8 ounces shall be removed from the container with a sampling thief. The granulation and particle size shall be determined on the sample thus obtained. Each container shall be individually tested. If any sample fails to comply with the requirements of Table II and Table III the container shall be rejected.
- 4.2.3.1.2 <u>Alternate sampling for granulation and particle size</u>. The composite sample prepared in accordance with 4.2.3.1 may be used in lieu of the samples required in 4.2.3.1.1. If

the composite samples fail to comply with the requirements in Tables II and III, the lot shall be rejected, or each container may be sampled in accordance with 4.2.3.1.1 for acceptance determination and if any container fails to comply with the requirements of Table II and III the container shall be rejected."

# PAGE 11

\* Delete paragraph 4.3.4.1.

Add the following paragraph:

- \* 4.3.6 Zinc. Change "ASTM Designation E34-58" to "ASTM E34".
- \* Delete paragraph 4.3.6.1

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\* 4.3.9 <u>Silicon as Si</u>. Change "ASTM Designation E34-58" to "ASTM E34".

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Add the following paragraphs.

- "4.3.11.2.1 Magnesium (alternate method) Code No. 20001.
- 4.3.11.2.1.1 Reagents. Chromic acid solution, approximately 0.1N as the acid. Dissolve 10.00 g of  $CrO_3$  in water and dilute to 100 ml. Magnesium standard solution. (One ml contains approximately 1.8 mg of magnesium). Dissolve 3 g ( $\pm$  0.5 mg) of pure magnesium in 125 ml of 1:4 hydrochloric acid. Dilute to 1000 ml in a volumetric flask. EDTA, standard solution, 0.1M. Dissolve 1.8 g of disodium dihydrogen ethyendiamintearaacetate dihydrate in distilled water and dilute to 1000 ml in a volumetric flask. Standardize against a 0.1M calcium or magnesium standard solution. Buffer solution, approximate pH of 10. Dissolve 65.5 g of reagent grade ammonium chloride in 300 ml of water. Add 570 ml of concentrated ammonium hydroxide and dilute to one liter. Erichrome Black T indicator solution. Dissolve 0.8 g of Erichrome Black T in a mixture of 40 ml of methanol and 60 ml of triethanolamine.
- 4.3.11.2.1.2 <u>Standardization</u>. Pipet 50.00 ml of magnesium standard into a 500 ml flask. Dilute to about 30 ml with distilled water. Add 20 ml of buffer solution and 6 10 drops of Erichrome Black T indicator. Titrate with EDTA to a color change of red to blue against a reflected-light background. There should be no red color remaining. Record the volume of EDTA consumed:

4.3.11.2.1.3 <u>Procedure</u>. Weigh approximately 1 g ( $\pm$  0.2 mg) of sample in a 250 ml beaker. Add 50 ml of 0.1 N chromic acid to the sample and allow to stand for 15 minutes, swirling the beaker every five minutes. Transfer the sample to a 30 ml, fine-porosity sintered glass crucible and wash the sample with 25 ml portions of distilled water 6 times. The last washing should be colorless. Wash the sample back into the original beaker with distilled water. Treat the crucible with two 10 ml portions 10 % hydrochloric acid to dissolve any metal remaining in it, returning the washings into the beaker distilled water. Cover with a watch glass. Add 10 ml of concentrated hydrochloric acid in small amounts to the sample. Heat to dissolve any remaining metal. Cool and transfer the solution to a 500 ml volumetric flask. Dilute to the mark with distilled water and mix. Pipet 50.00 ml of magnesium standard into a 500 ml flask. Dilute to about 300 ml with distilled water. Add 20 ml of buffer solution and 6 to 10 drops of Erichrome Black T indicator. Titrate with EDTA to a color change of red to blue against a reflected light background. There should be no red color remaining. Record the volume of EDTA consumed and calculate.

% metallic magnesium =  $\frac{\text{ml of EDTA x mg Mg per ml x 100}}{\text{grams of sample in aliquot x 100}}$ 

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- 4.3.17 Ellipsoidal Type IV. Delete in its entirety and substitute the following:
- "4.3.17 Ellipsoidal Type IV. Code Number 19001. Microscopic Analysis. Remove approximately a 10 gram sample from the top, middle and bottom strata of the quart jar, and then place the sample into a container, cover, and tumble the container for five minutes. With a scoop, remove a ¼ gram sample from the container and sprinkle the sample on a microscopic slide. The slide shall be placed on a stereo microscope (magnification may be 16 x to 24 x range) equipped with an eyepiece micrometer (photographic). Light shall be transmitted through the top and bottom of the sample. The length and width of 100 particles shall be measured. For each particle, the length shall be divided by the width to obtain the L/W oblong shape ratio. The percentage of ratio above 1.5 % shall be determined."
- 4.3.18 Nitrogen. Delete in its entirety.

# PAGE 18

Add the following paragraph:

"6.1.1 <u>Type V aluminum powder</u>. The type V aluminum powder outlined in this specification is intended for use in the rocket motor of the 105MM M913.

**NOTE:** The margins of this amendment are marked with an asterisk or vertical line to indicate where changes (additions, modifications, corrections, deletions) from the previous were made. This was done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in these notations. Bidders and contractors are cautioned to evaluate the requirements of this document based on the entire content irrespective of the marginal notations and relationship to the last previous amendment.

CUSTODIAN: PREPARING ACTIVITY: ARMY-AR DLA-GS

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ARMY-AR

Project: 6810-1641.